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भारतीय मानक एल्जिनिक अम्ल, खाद्य ग्रेड — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

ALGINIC ACID, FOOD GRADE — SPECIFICATION

(First Revision)

(Incorporating Amendment No. 1)

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the expansion of processed food industry, food additives, generally in small quantities, are increasingly used to improve the appearance, flavour, texture or storage properties, etc of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. This standard has been prepared to cover purity and identification of the substances. The standard would help in checking purity which is required to be checked at the stage of manufacture, for it is difficult to detect the impurity once the additive has been added to the processed foods. Besides, the standard is intended to guide the indigenous manufacturers in making their product conform to the specification that is accepted by scientists, health authorities and international bodies.

Use of alginic acid as stabilizer, thickener, and emulsifier has been permitted for certain foods in the *Prevention of Food Adulteration Rules*, 1955.

Chemical Name

Alginic acid. Its emprical formula is $(C_6H_8O_6)n$, its equivalent weight calculated is 176.27 and actual (avg) is 200.00.

This standard was first published in 1975. It is being revised to take into account the latest technological developments in the field, and also to bring it in a line with the International Standards.

In the preparation of this standard the requirements given in the following publications have been taken into consideration:

Food Chemical Codex published by the National Academy of Sciences and National Research Council, Washington DC, USA.

FAO Food and Nutrition Papers No. 4 — 'Specifications for the identity and purity of thickening agents, anticaking agents, antimicrobials, antioxidants and emulsifiers; published by the joint FAO/WHO Expert Committee on Food Additives, Rome, 1978.

EEC Directive No. 78/663/EEC dated 25 July 1978 laying down specific criteria for purity for emulsifiers, stabilizers, thickners and gelling agent for use in foodstuffs.

This edition 2.1 incorporates Amendment No. 1 (January 1994). Side bar indicates modification of the text as the result of incorporation of the amendment.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 1960 'Rules for rounding off numerical values (<code>revised</code>)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ALGINIC ACID, FOOD GRADE — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the method of sampling and test for alginic acid, food grade.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard.

IS~No.	Title		
1070 : 1992	Reagent grade water ($third\ revision$)		
1699 : 1974	Methods of sampling and test for food colours (first revision)		
2557 : 1963	Annatto colour for food products		
5887 (Part 1): 1976	Methods for detection of bacteria responsible for food poisioning: Part 1 Isolation, identification and enumeration of <i>Escherichia coli</i> (<i>first revision</i>)		
5887 (Part 2): 1976	Methods for detection of bacteria responsible for food poisioning: Part 2 Isolation, identification and enumeration of Staphylococcus aureus and faecal streptococci (first revision)		

3 REQUIREMENTS

3.1 Description

Alginic acid shall be the hydrophilic colloidal carbohydrate extracted by the use of dilute alkali from various species of brown seaweed (Phaeophyceae). It mav be described glycuronoglycan chemically as a linear mainly of B consisting (1-4)D-mannuronic and L-guluronic acid units in the pyranose ring forms. It occurs as a white to yellowish-white, fibrous powder. It shall be odourless and tasteless.

3.2 Identification Tests

3.2.1 Solubility

Insoluble in water and organic, solvents; slightly soluble in solutions of sodium carbonate, sodium hydroxide and trisodium monophosphate.

3.2.2 pH - 2.0 - 3.4 (3 in 100 suspension).

- **3.2.3** To a 0.5 percent solution of the sample in sodium hydroxide add one-fifth of its volume of a 2.5 percent aqueous solution of calcium chloride. A voluminous, gelatinous precipitate is formed. This test distinguishes alginic acid from arabic gum, carboxymethyl cellulose, carboxymethyl starch carrageenan, gelatine, ghatti gum, karaya gum, locust bean gum, methyl cellulose, pectin and tragacanth.
- **3.2.4** To a 0.5 percent solution of the sample in sodium hydroxide add one-half of its volume of a saturated solution of ammonium sulphate. No precipitate is formed. This test distinguishes alginic acid from agar, carboxymethyl cellulose, carrageenan, de-esterified pectin, gelatine, locust bean gum, methyl cellulose and strach.
- **3.2.5** Dissolve as completely as possible $0.01~\rm g$ of the sample by shaking with $0.15~\rm ml$ of $0.1~\rm N$ sodium hydroxide, and add $1~\rm ml$ of acid ferric sulphate. Within $5~\rm minutes$, a cherry-red colour develops that finally becomes deep purple.
- **3.3** The material shall also conform to the requirements given in Table 1.

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be filled in containers with as little air space as possible. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

Table 1 Requirements for Alginic Acid, Food Grade

(Clause 3.3)

	Sl	Characteristic	Requirement	Method of Test, Ref to	
]	No.			Annex of this Standard	Other Indian Standards
	(1)	(2)	(3)	(4)	(5)
	i)	Purity, as $(C_6H_8O_6)n$, percent by mass, Min	91	A-1	_
	ii)	Moisture, percent by mass, on drying at 105°C for 4 h, Max	15	A-2	_
	iii)	In soluble matter, percent by mass, Max	0.2	A-3	_
	iv)	Ash (on dry basis), percent by mass, ${\it Max}$	4	A-4	_
	v)	Acid insoluble ash (on dry basis), percent by mass, ${\it Max}$	0.5	A-5	_
	vi)	Lead (as Pb), mg/kg, Max	10	_	9 of IS 1699 : 1974
,	vii)	Arsenic (as As), mg/kg, Max	3	_	10 of IS 1699 : 1974
v	riii)	Heavy metals (as Pb), mg/kg, Max	40	A-6	_
	ix)	E. Coli	Absent (in 1 g)	_	IS 5887 (Part 1) : 1976
	x)	Salmonella	Absent (in 10 g)	_	IS 5887 (Part 3) : 1976

4.3 Marking

Each container shall be marked legibly to give the following information:

- a) Name of the material including the words 'Food Grade';
- b) Source of manufacture;
- c) Minimum net mass or content;
- d) Batch or code number;
- e) Date of manufacture; and
- f) Any other details required under the Standards of Weights and Measures (Packaged Commodities) Rules, 1977/Prevention of Food Adulteration Rules, 1955.

4.3.1 The container may also be marked with the Standard Mark.

5 SAMPLING

5.1 The representative samples of the material shall be drawn according to the method prescribed in Appendix A of IS 2557: 1963.

6 TESTS

6.1 Tests shall be carried out as prescribed in **3.2** and col 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see~ IS 1070:1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

ANNEX A

(Table 1)

METHOD OF TEST FOR ALGINIC ACID

A-1 PURITY

A-1.1 Apparatus

The apparatus required shall be as shown in Fig. 1. It consists essentially of a soda-lime column A, a mercury valve B connected through a side tube C to a reaction flask D by means of a rubber connection. D is a 100-ml round-bottomed, long-necked boiling flask with a 24/40 ground-glass joint attached. The oilbath E shall be maintained at $145^{\circ}\mathrm{C}$ by means of a thermoregulator and an immersion heater.

The reaction flask shall be provided with a 20-cm reflux condenser F terminating in a trap G containing 25 g of 20-mesh zinc or tin, which can be connected with an absorption flask H (a 250-ml Erlenmeyer flask equipped with a 24/40 ground-glass joint and a side tube attached a little below the ground-glass joint, as shown in Fig. 1).

Flask H shall be provided with an absorption tower *I* the lower part of which consists of an 18-mm tube fitted with a medium porosity fritted borosilicate-glass disc sealed to the inner part of the lower end of a 24/40 ground-glass joint and terminating 1 or 2 mm above the bottom of the absorption flask when the joint is in place. A trap consisting of a bulb of approximately 100-ml capacity, is blown above the ground portion of the joint, and the outer portion of a 24/40 ground-glass joint shall be sealed on above this bulb. The absorption tower, from bottom of the disc to the top of ground-glass joint, is approximately 30 cm in length. The top of the tower is fitted with a hollow ground-glass stopper with a short side tube attached. The tower assembly may be attached to a soda lime tower J connected with a water pump by means of a capillary-tube regulator K which serves to sweep 1700 to 2 000 ml of carbon dioxide-free air per hour through the apparatus during the heating period.

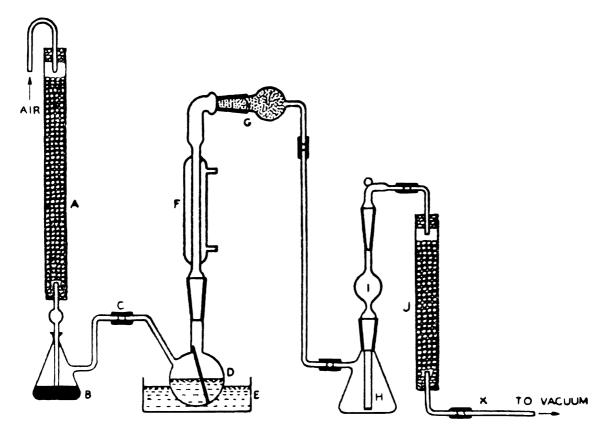


Fig. 1 Apparatus for Determination of Alginic Acid

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A-1.2 Reagents

A-1.2.1 Hydrochloric Acid — 19 percent and $0.1~\mathrm{N}.$

A-1.2.2 *Sodium Hydroxide* — 0.25 N.

A-1.2.3 *n-Butanol*

A-1.2.4 Barium Chloride Solution — Dissolve 12 g of barium chloride (BaCl $_2$. 2H $_2$ O) in sufficient water to make 100 ml.

A-1.2.5 *Phenolphthalein Solution* — Dissolve 1 g of phenolphthalein in 100 ml of alcohol.

A-1.3 Procedure

Transfer about 250 mg of the sample, previously dried at 105°C for 4 hours accurately weighed, to the reaction flask D, add 30 ml of 19 percent hydrochloric acid, insert a small boiling tube, and connect it to the reflux condenser F using syrupy phosphoric acid as lubricant. (Stopcock grease may be used for other connections. Draw a current of carbon dioxide-free air through the entire assembly for about 10 minutes and then discontinue it. Disconnect the absorption tower I, rapidly transfer from a pipette 25.0 ml of 0.25 N sodium hydroxide into the absorption flask H, add 5 drops of n-butanol, and again attach it to the absorption tower. Raise them oil-bath E, previously heated to 145 ± 2°C, until the oil level is several millimetres above the liquid level in the reaction flask. After the initial rapid evolution of carbon dioxide has subsided, resume the passage of carbon dioxide-free air through the apparatus and continue the heating at about 145°C for two hours. At the end of the 2-hour period discontinue the current of air, and disconnect the absorption flask H and the lower part of the absorption tower *I* from the rest of the assembly. Remove the absorption tower unit, washing any adhering sodium hydroxide solution into the flask with several small portions of water. To the flask add 10 ml of 10 percent barium chloride solution, stopper the flask, shake gently for about 2 minutes, add phenolphthalein and titrate with 0.1 N hydrochloric acid. Perform a blank determination and make any necessary correction. Each millilitre of 0.25 N sodium hydroxide consumed in the assay is equivalent to 5.5 mg of carbon dioxide or to 25 mg of alginic acid.

A-2 MOISTURE

A-2.1 Apparatus

A-2.1.1 Oven — electric, maintained at $105^{\circ} \pm 1^{\circ}$ C.

A-2.1.2 Weighing Bottle — glass-stoppered, shallow.

A-2.2 Procedure

Weigh accurately about 10 g of the well-mixed material in a tared weighing bottle. Distribute the material as evenly as practicable to a depth of about 5 mm. Place the bottle containing the material (uncovered) in the oven maintained at $105^{\circ} \pm 1^{\circ}$ C. Remove the bottle from the oven after four hours, close the bottle promptly and allow it to come to room temperature in a desiccator. Weigh it.

A-2.2.1 Calculation

Moisture, percent by mass =
$$\frac{100 \times (M_1 - M_2)}{(M_1 - M)}$$

where

M = mass, in g, of the empty bottle;

 M_1 = mass, in g, of the bottle with the material before drying;

 M_2 = mass, in g, of the bottle with the material after drying and after it has come to room temperature.

A-3 INSOLUBLE MATTER

A-3.1 Transfer about 1 gram of the material, accurately weighed, into a 600-ml beaker, add 100 ml of 0.1 N sodium hydroxide and dilute the mixture to 200 ml. Cover the beaker, heat to boiling and boil gently for one hour with frequent stirring. Filter while hot through a tared Gooch crucible provided with an asbestos mat, wash thoroughly with hot water, dry at 105°C for one hour, cool and weigh.

A-4 ASH

A-4.1 Weigh accurately about 3 g of the material previously dried at 105°C for 4 hours, in a tared crucible and incinerate at a low temperature, not exceeding a dull red heat until free from carbon. Cool the crucible and its content in a desiccator, weigh, and determine the weight of ash.

A-5 ACID INSOLUBLE ASH

A-5.1 Procedure

Boil the ash obtained under A-4 with 25 ml of diluted hydrochloric acid for 5 minutes. Collect the insoluble matter on a tared Gooch crucible or ashless filter paper. Wash with hot water. Ignite and weigh. Calculate the percentage of acid insoluble ash from the weight of sample taken.

A-6 HEAVY METALS

A-6.1 Reagents

A-6.1.1 Ammonia Solution

Dilute 400 ml of ammonium hydroxide (28 percent) to 1 000 ml with water.

A-6.1.2 *Hydrochloric Acid* — 10 percent

A-6.1.3 Lead Nitrate Stock Solution

Dissolve 159.8 mg of lead nitrate in 100 ml of water containing 1 ml of nitric acid. Dilute with water to 1 000 ml and mix. Prepare and store the solution in lead free glass containers.

A-6.1.4 Standard Lead Solution

Dilute 10 ml of lead nitrate stock solution, accurately measured, with water to 100 ml. Each ml of the solution so prepared contains the equivalent of $10 \mu g$ of lead ion (Pb). Prepare the solution on the day of use.

A-6.1.5 *Nitric Acid* — 10 percent (v/v)

A-6.1.6 Sulphuric Acid — 94.5 to 95.5 percent (v/v).

A-6.1.7 Acetic Acid — 6 percent (m/v).

A-6.1.8 Hydrogen Sulphide

A saturated solution of hydrogen sulphide made by passing H_2S in cold water.

A-6.2 Procedure

A-6.2.1 Solution A

Take 2 ml of the standard lead solution in a 50-ml Nessler tube and add 23 ml of water.

Adjust the pH to between 3.0 and 4.0 by addition of acetic acid or ammonia solution. Dilute with water to 40 ml and mix.

A-6.2.2 *Solution B*

Place 500 mg of the sample, accurately weighed in a suitable crucible. Add sufficient nitric acid to wet the sample, and carefully ignite at a low temperature until thoroughly charred, covering the crucible loosely with a suitable lid during ignition. After the substance is thoroughly carbonized, add 2 ml of nitric acid and 5 drops of sulphuric acid and cautiously heat until white fumes are evolved. Then ignite, preferably in a muffle furnace, at 500 to 600°C until the carbon is all burnt off. Cool, add 4 ml of dilute hydrochloric acid, cover and digest on a steam bath for 10 to 15 minutes. Uncover and slowly evaporate on a steam bath to dryness. Moisten the residue with one drop of hydrochloric acid, add 10 ml of hot water and digest for 2 minutes. Add, dropwise, ammonia solution until the solution is just alkaline to litmus paper. Dilute with water to 25 ml and adjust the pH to between 3.0 and 4.0 (pH indicator paper) by the addition of dilute acetic acid. Filter if necessary. Wash the crucible and the filter with 10 ml of water. Transfer to a 50-ml Nessler tube. Dilute the combined filtrate and washing with water to 40 ml and

A-6.2.3 To each tube add 10 ml of freshly prepared hydrogen sulphide. Mix and allow to stand for 45 minutes and view down over a white surface. The colour of solution B shall not be darker than that of solution A.

Standard Mark The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. FAD 8 (0099).

Amendments Issued Since Publication

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AMENDMENT NO. 2 FEBRUARY 2005 TO IS 7928: 1993 ALGINIC ACID, FOOD GRADE — SPECIFICAITON

(First Revision)

(Page 1, clause 2.1) — Insert reference of the following Indian Standards at the appropriate place:

IS No.	Title .		
'5402 : 2002	General guidance for enumeration of micro- organisms — Colony count technique at 30°C (first revision)		
5403 : 1999	Method for enumeration of yeast and mould count in foodstuffs and animal feeds (first revision)'		

[Page 2, Table 1, Sl No. (i), col 2] — Substitute 'Purity, as (C6H8O6)n, percent by mass, on dry basis, Min' for 'Purity, as (C6H8O6)n, percent by mass, Min'.

[Page 2, Table 1, Sl No. (vi), col 3] — Substitute '5' for '10'.

(Page 2, Table 1) — Insert the following at the end of the table:

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of This Standard	Other Indian Standards
xi)	Total plate count per g, Max	5 000	******	5402 : 2002
xii)	Yeasts and moulds per g, Max	500		5403 : 1999